UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

Analytical Chemistry Section
Building 402, ARC-East
Beltsville, Maryland 20705

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

August 7, 1984

MEMORANDUM

SUBJECT: FAP#2H5357 Ethephon on Wheat and Barley Method Trial

TO: Charles L. Trichilo, Chief
Residue Chemistry Branch
Hazard Evaluation Division (TS-769)

THRU: Donald A. Marlow, Chief
Chemical Operations Branch

THRU: Warren R. Bontoyan, Section Head
Analytical Chemistry Section

We were requested by Chemistry Branch to conduct a method trial on Ethephon produced by Union-Carbide Company. The petitioner's method entitled "Detailed Method of Analysis for Residues of (2-Chloroethyl) Phosphonic Acid (Ethephon) in wheat, oat and barley grain, straw and milling fractions" is to be used for the trial. Wheat straw were fortified with 0, 0.02, 0.10 and 2.0 ppm ethephon. The method is poorly written and not clear in several steps given in the method. However, the method appeared satisfactory at 2.0 ppm level of fortification. Wheat straw was supplied by USDA Beltsville, Maryland.

In the method, Ethephon is extracted from the wheat straw by 1% methanol citric acid solution in a Soxhlet extractor. Transfer extract and methanol rinses to a 250 ml glass-stoppered graduated cylinder. Keep the total volumn not more than 150 ml.

Pipet 1/10 of the final volume to a 15 ml graduated centrifuge tube and concentrate to 1.5 ml using a gentle stream of nitrogen and a 30-35° C bath. Add 0.5 ml "10%" methanolic HCL, 8 ml diethyl ether, mix well and let stand for 10 minutes. Decant the supernatant to a clean, graduated centrifuge tube. Again concentrate it to 1 - 1.2 ml by stream of dry nitrogen. Add diazomethane solution until a permanent yellow color is obtained. Concentrate and centrifuge once more. Pass the liquid through a deactivated florisil column. Collect the residue from the florisil

by acetone. Concentrate the eluate to 1.0 ml. This solution representing 0.5 gms straw/ml, is now ready for gas chromatographic analysis.

Cas Chromatograph: Hewlett-Packard 5840A equipped with FPD/P filter detector.

Column: 6' x 2mm I.D. glass packed with 6% FFAP on 60/80 mesh chromosorb G, AW, DMCS

Tempertures: Column: 165
Inlet: 200
Detector: 250

Carriergas (He) 35-40 ml/min; Hydrogen 50 ml/min; 4 ng dimethylethephon standard eluted in 7 min and has 50% FSD at attn 2.

Results:

	Ethephon Fortified (ppm)	Recovered (ppm)	
	0.0	<.05	
	0.0	<.05	
*	0.02	-	
	0.02	-	
*	1.0	-	
	1.0	-	
	2.0	1.74	87%
	2.0	1.56	78%

* fortification were too low for meanful gas chromatography measurements.

Comments

(1) Union carbide supplied ethephon and its dimethyl derivative standards. Dimethyl ethephon is stable at least 2 months at room temperature. It should be stable to be held in a repository in its neat state.

(2) Each ng ethephon is equivalent to 0.84 ng dimethyl ethephon.

(3) In the beginning of the validation, we had considerable amount of diffficulty to bring the 6% FFAP GC column up to the needed level of column efficiency. When we informed the company about this difficulty, we were asked to prepare the dimethyl ethephon standard in 1% methanolic dimethyltartrate solution to "stabilize" the column. We did improve the efficiency of the column after injecting several standards solutions and conditioning the column at 220°C over night. However, we could not get better then 50% FSD response for a 4 ng

standard injection.

(4) All samples were yellow in color and had some solid materials prior to the methylation step. We added 5-6 ml diazomethane solution to each of the sample.

(5) Union Carbide do not use 20% OV-11 GC column for their ethephon work. We tried this column and it was not suitable for the trial.

(6) Once the GC work is done it will take two days to analyze one set of samples.

(7) Method should state the preparation of dimethylethephon standard in 1% dimethyl tartrate emthanolic solution which is used for the condition of the GC column. It should also state that GC column is 6' x 2 mm ID.

King Zee, Chemist
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